

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ :	A1	(11) International Publication Number: WO 96/29980 (43) International Publication Date: 3 October 1996 (03.10.96)
A61K 7/50		
(21) International Application Number:	PCT/EP96/01167	(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).
(22) International Filing Date:	15 March 1996 (15.03.96)	
(30) Priority Data:	9506038.0 24 March 1995 (24.03.95) GB	
(71) Applicant (for AU BB CA GB IE KE LK LS MN MW NZ SD SG SZ TT UG only):	UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB).	
(71) Applicant (for all designated States except AU BB CA GB IE KE LK LS MN MW NZ SD SG SZ TT UG):	UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).	
(72) Inventors:	HAGUE, Jonathan, David; 24 Trafalgar Drive, Bebington, Wirral, Merseyside L63 7RW (GB). KHAN-LODHI, Abid, Nadim; 20 Beech Grove, Hoole, Chester, Cheshire CH2 3HJ (GB). REID, Euan, Stuart; 4 Meadoway, Bebington, Wirral, Merseyside L62 2AR (GB).	
(74) Agent:	ROTS, Maria, J., F.; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB).	

(54) Title: HAIR CONDITIONING COMPOSITION

(57) Abstract

A transparent conditioning composition for hair comprises a stable transparent dispersion of: (i) a substantially water-insoluble quaternary ammonium material having two C₁₂₋₂₈ alkyl or alkenyl groups connected to the quaternary ammonium head group via at least one ester linkage; and (ii) a solubilising agent comprising at least one surfactant, preferably cationic surfactants.

BEST AVAILABLE COPY

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

HAIR CONDITIONING COMPOSITION**TECHNICAL FIELD**

5 This invention relates to hair conditioning compositions, more particularly to hair conditioning compositions which are transparent.

BACKGROUND AND PRIOR ART

10

Hair conditioning compositions may be formulated to select those components which together impart a desirable combination of properties. Typically such compositions contain a mixture of substantially water-insoluble hydrocarbon-based conditioning agents, examples being fatty alcohols and acids, long chain dialkyl quaternary ammonium salts, fatty tertiary amine salts, paraffin waxes and glycerol fatty acid esters, dispersed in water, often also in the presence of a water-soluble long chain cationic surfactant. The total solids content of such products is usually between 3 and 10%, and the conditioning agents generally form a mixed morphology comprising a dispersion of stacked lamellar structures and solid fatty particles.

25

Compositions of the type described above offer very effective detangling of wet hair, and impart other desirable properties to hair such as ease of dry combing, smoothness both wet and dry, softness in the dry state, and lack of flyaway due to reduction of the static charge generated on hair. This combination of properties is generated to a greater degree by the molecular organisation of components in the lamellar structures.

35 Such compositions are, however, invariably opaque since both the lamellar structures and fatty particles are

present at the super-micron size range, and so scatter light very effectively. However, hair conditioning compositions are often desired to be transparent, i.e. optically clear or substantially clear. This is of benefit as regards visual product appeal to the consumer.

For people whose hair is naturally greasy, conventional opaque conditioners are also often undesirable because of heavy coating and residue build up on the hair and excessive conditioning perceptions. It is believed that excessive levels of hydrocarbon-based conditioning agents in conditioning compositions contribute to the sensory negatives of heaviness and greasiness.

There is thus a desire to provide a transparent conditioning composition which is lower in its levels of substantially insoluble hydrocarbon based conditioning agents, so minimising the problems associated with high levels of fatty material. There is equally the requirement, however, to ensure that performance attributes of the composition in terms of the desirable hair conditioning properties referred to above are not reduced as a consequence.

One particular known hair conditioning composition which is transparent is disclosed in JP-A-52-122638 and comprises a cationic surfactant component, particularly cetyltrimethylammonium chloride, and a phosphoric acid ester component, particularly mono/diphosphate ester of C18 (3EO) alcohol. However, such compositions have been recognised as having limited hair conditioning properties. It has also been found that it is not possible to simply increase the proportion of phosphoric acid ester component with the aim of enhancing the hair conditioning benefits imparted by the composition, since that results in a cloudy product, which, as discussed, is undesirable.

EP-A-0100164 discloses hair conditioning preparations comprising anionic surfactant and a cationic polymer, rendered clear by inclusion of a clarifying agent. When diluted with water upon use, an insoluble complex is
5 formed between the cationic polymer and anionic surfactant, which is precipitated and thereby deposited on the hair. Such preparations typically deposit material very heavily onto the hair, which can lead to dulling effects and greasy build-up.

10 US 4744977 discloses hair conditioning compositions comprising particular β -hydroxy quaternary ammonium compounds. These hair conditioning compounds are indicated as being more compatible with high-foam anionic
15 surfactants compared with other known quaternary ammonium compounds; giving products which are less cloudy and less prone to precipitation of insoluble deposits. The reference discloses generally a wide variety of anionic surfactants for use in combination with the defined
20 cationic surfactant in hair conditioning shampoos.

EP-A-571086 discloses hair care compositions comprising an ester-linked quaternary ammonium compound, and possibly also a cationic surfactant as a conditioning agent. There
25 is no disclosure of a transparent dispersion of such materials.

EP-A-345842 discloses a fabric softener comprising a quaternized ester-ammonium compound and a substituted
30 imidazoline ester in the form of sub-micron size dispersions in liquid carrier. Optional ingredients include surfactants. There is no disclosure of a hair conditioners in the form of a transparent dispersion.

35 EP-A-309052 discloses a fabric softener comprising a quaternized ester-amine and a linear alkoxylated alcohol

in the form of a sub-micron size dispersion in liquid carrier. Optional surfactant may be included. There is no disclosure of a hair conditioner in the form of a transparent dispersion.

5

US 4370272 discloses novel quaternary ammonium compounds useful, inter alia, in hair conditioners. There is no disclosure of a hair conditioner in the form of a transparent dispersion.

10

We have now found that hair conditioning compositions can be prepared which are optically clear or transparent without the need for inclusion of clarifying agents, transparency materials or heavily-flocculating polymer-surfactant mixtures. The present invention can provide hair conditioning compositions having excellent optical properties, despite containing principally water-insoluble hair conditioning agents, and which furthermore deposit/build up perceptibly less material to the hair surface relative to conventional opaque conditioners, but with no concurrent loss of desired conditioning performance.

15

20

According to one aspect of the invention there is provided a hair conditioning composition comprising a transparent dispersion of:

30

(i) a hair conditioning compound comprising a substantially water-insoluble quaternary ammonium material having two C₁₂₋₂₈ alkyl or alkenyl groups connected to the quaternary ammonium head group via at least one ester linkage , and;

35

(ii) a solubilising agent comprising at least one surfactant selected from anionic, amphoteric, nonionic, and cationic surfactants or mixtures thereof.

5 DETAILED DESCRIPTION OF THE INVENTION

Without wishing to be bound by theory it is believed that the hair conditioning composition of the invention is not in conventional lamellar form, and in water may be 10 solubilised at least partially by the action of the surfactant to form self-size-limiting molecular aggregates, such as micelles or micellar solutions with solid or liquid interiors or mixtures thereof. These can be considered as particles dispersed in a continuous 15 phase. The aggregates have a predictable small particle size (less than 0.1 micron) which is not affected by processing, resulting in production of an aqueous dispersion that is transparent and also has good hair conditioning properties. It is thought that it is this 20 new structure of the hair conditioning compositions of the invention that overcomes the problems of the prior art.

The dispersion is generally an aqueous dispersion.

25 When the hair conditioning composition is formulated with water to a concentration of 1 wt% of the hair conditioning compound (i), the compound (i) is substantially present in solution, that is, at least about 70 wt% and preferably 80 wt% of compound (i) is in solution.

30 The transparent dispersion is found to be stable over an extended period of time, of many months at least, so that the compositions of the invention have a reasonable shelf life.

35

THE HAIR CONDITIONING COMPOUND

The hair conditioning compounds used in the compositions of the invention are molecules which provide excellent hair conditioning, characterised by chain melting - $L\beta$ to $L\alpha$ - transition temperature greater than 25°C, preferably greater than 35°C, most preferably greater than 45°C. This $L\beta$ to $L\alpha$ transition can be measured by DSC as defined in "Handbook of Lipid Bilayers", D. Marsh, CRC Press, Boca Raton Florida, 1990 (Pages 137 and 337).

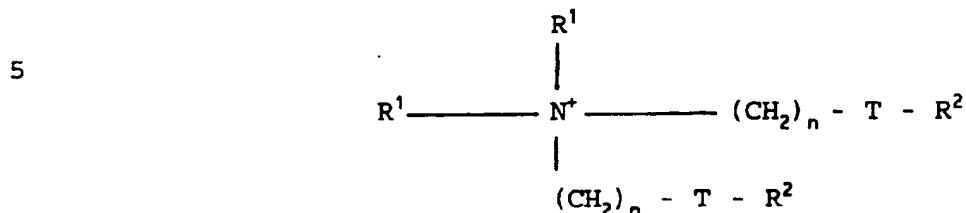
Substantially insoluble hair conditioning compounds in the context of this invention are defined as hair conditioning compounds having a solubility less than 1×10^{-3} wt% in demineralised water at 20°C. Preferably the hair conditioning compounds have a solubility less than 1×10^{-4} . Most preferably the hair conditioning compounds have a solubility at 20°C in demineralised water from 1×10^{-8} to 1×10^{-6} . However, when solubilised by the action of the solubilising agent, the hair condition compounds can form a transparent, small particle size dispersion.

It is preferred if the alkyl or alkenyl groups of the hair conditioning compound are predominantly linear.

Preferably the hair conditioning compound of the invention has two long chain alkyl or alkenyl chains with an average chain length equal to or greater than C_{14} . More preferably each chain has an average chain length equal to or greater than C_{16} . Most preferably at least 50% of each long chain alkyl or alkenyl group have a chain length of C_{18} .

It is preferred if the hair conditioning compound is a water insoluble quaternary ammonium material which comprises a compound having two C_{12-18} alkyl or alkenyl groups connected to the molecule via at least one ester link. It is more preferred if the quaternary ammonium material has two ester links present.

A preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula:

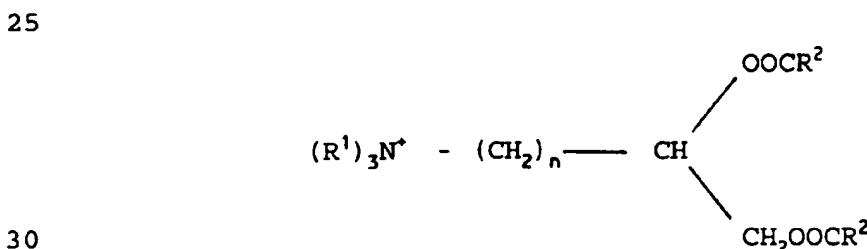


10 wherein each R¹ group is independently selected from C₁₋₄ alkyl, hydroxyalkyl or C₂₋₄ alkenyl groups; and wherein each R² group is independently selected from C₈₋₂₈ alkyl or alkenyl groups;

15 each T independently represents - O - C = , - O - C = O -
 , or - C = O - ;

20 and n is an integer from 0 to about 5.

An especially preferred type of quaternary ammonium material can be represented by the formula:



30 wherein R¹, n and R² are as defined above.

It is advantageous for environmental reasons if the
35 quaternary ammonium material is biologically degradable.

Compounds of this formula suitable for use in the hair conditioning compositions of the invention and their method of preparation are, for example, described in US 4 137 180 (Lever Brothers), the disclosure of which is 5 incorporated herein by reference.

A particularly preferred example is 1,2 - ditallowoyloxy - 3 - trimethylammonium propane chloride.

10 The amount of the hair conditioning compound in the composition of the invention is suitably in the range of from about 0.01 to about 20 wt% based on total weight of the composition, more preferably from 0.05 to 10 wt%, even more preferably from 1 to 2 wt%. It is generally 15 preferred not to use more than 2% of the hair conditioning compound, as higher concentrations may adversely affect turbidity.

THE SOLUBILISING AGENT

20 The solubilising agent comprises at least one surfactant selected from anionic, amphoteric, nonionic, and cationic surfactants or mixtures thereof.

25 These may be characterised in terms of their phase behaviour. Suitable solubilising agents are surfactants for which, when contacted with water, the first lyotropic liquid crystalline phase formed is normal cubic (I1) or normal cubic-bicontinuous (V1) or hexagonal (H1) or 30 nematic (Nel), or intermediate (Int1) phase as defined in the article by G J T Tiddy et al, J Chem Soc. Faraday Trans. 1. , 79, 975, 1983 and G J T Tiddy, "Modern Trends of Colloid Science in Chemistry and Biology", Ed. H-F Eicke, 1985 Birkhauser Verlag Basel. Surfactants forming 35 L_a phases at concentrations of less than 20 wt% are less suitable.

Suitable anionic surfactants are the alkyl sulphates, alkyl ether sulphates, alkaryl sulphonates, alkyl succinates, alkyl sulphosuccinates, N-alkoyl sarcosinates, alkyl phosphates, alkyl ether phosphates, alkyl ether carboxylates, and alpha-olefin sulphonates, especially their sodium, magnesium, ammonium and mono-, di- and triethanolamine salts. The alkyl groups generally contain from 8 to 18 carbon atoms and may be unsaturated. The alkyl ether sulphates, alkyl ether phosphates and alkyl ether carboxylates may contain from 1 to 10 ethylene oxide or propylene oxide units per molecule, and preferably contain 2 to 3 ethylene oxide units per molecule.

Examples include sodium lauryl sulphate, triethanolamine lauryl sulphate 1EO, 2EO and 3EO, ammonium lauryl sulphate and ammonium lauryl ether sulphate 1EO, 2EO and 3EO.

Suitable amphoteric surfactants include alkyl amine oxides, alkyl phosphine oxides, alkyl sulphoxides, alkyl betaines, alkyl amidopropyl betaines, alkyl sulphobetaines, alkyl glycicates, alkylcarboxyglycimates, alkyl amphopropionates, alkyl amphoglycimates, alkyl amidopropyl hydroxysultaines, acyl taurates and acyl glutamates, wherein the alkyl and acyl groups have from 8 to 19 carbon atoms. Examples include lauryl amine oxide, cocodimethyl sulphopropyl betaine and preferably lauryl betaine, cocamidopropyl betaine and sodium cocamphopropionate.

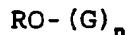
Nonionic surfactants include condensation products of aliphatic (C_{8-18}) primary or secondary linear or branched-chain alcohols or phenols with alkylene oxides, usually ethylene oxide, and generally having from 6 to 30 ethylene oxide groups.

10

Other suitable nonionics include mono- or di-alkyl alkanolamides. Examples include coco mono- or diethanolamide and coco mono-isopropanolamide.

5 Further suitable nonionic surfactants are the alkyl polyglycosides (APGs). Typically the APG is one which comprises an alkyl group connected (optionally via a bridging group) to a block of one or more glycosyl groups. Preferred APGs are defined by the following formula:

10



wherein R is a branched or straight chain alkyl group which may be saturated or unsaturated and G is a
15 saccharide group.

R may represent a mean alkyl chain length of from about C₅ to about C₂₀. Preferably R represents a mean alkyl chain length of from about C₈ to about C₁₂. Most preferably the
20 value of R lies between about 9.5 and about 10.5. G may be selected from C₅ or C₆ monosaccharide residues or mixtures of C₅ and C₆ monosaccharide residues, and is preferably a glucoside. G may be selected from the group comprising glucose, xylose, lactose, fructose, mannose and
25 derivatives thereof. Preferably G is glucose.

The degree of polymerisation , n, may have a value of from about 1 to about 10 or more. Preferably, the value of n lies in the range of from about 1.1 to about 2. Most
30 preferably the value of n lies in the range of from about 1.3 to about 1.5.

Suitable alkyl polyglycosides for use in the invention are commercially available and include for example those
35 materials identified as ORAMIX NS10 ex Seppic; PLANTAREN 1200 and PLANTAREN 2000 ex Henkel.

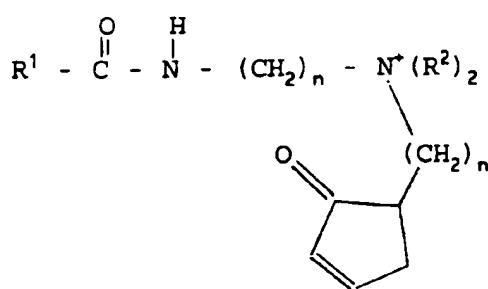
Silicone-based nonionic or cationic surfactants such as DC 190 ex Dow Corning (nonionic) may also be used.

5 Cationic surfactants are particularly preferred solubilising agents according to the invention.

Preferred types of cationic surfactants for use in the invention are quaternary ammonium hydroxides or salts thereof, e.g. halides. Examples of suitable cationic surfactants for use in the invention include:

15 tetramethylammonium salts, tetraethylammonium salts,
cetyltrimethylammonium salts, cetylpyridinium chloride,
octyltrimethylammonium salts, dodecyltrimethylammonium
salts, hexadecyltrimethylammonium salts,
octyldimethylbenzylammonium salts,
decyldimethylbenzylammonium salts,
stearyldimethylbenzylammonium salts,
oleyldimethylbenzylammonium halides, methyl bis-(-2-
20 hydroxyethyl) oleyl ammonium chloride, oleyl ammonium
chloride, tallow trimethylammonium salts,
cocotrimethylammonium salts and polyethoxylated quaternary
ammonium salts. Preferred ammonium salts are the chlorides
and bromides.

25 Further suitable cationic surfactants include those identified by the CTFA definitions Quaternium-5, Quaternium-31, Quaternium-18, and salts, eg chloride, of the quaternary ammonium derivative of formula:



in which R¹ represents C₁₂₋₂₂ alkyl, preferably C₁₈ alkyl, each R² independently represents C₁₋₄ alkyl, and n is an integer from 1 to about 5.

5

Other surfactants which under appropriate pH conditions have cationic character can also be used, for example amidoamine derivatives of lauric acid and alkyl betaines.

10 Mixtures of any of the foregoing materials may also be suitable. A particularly preferred cationic surfactant for use in the invention is cetyltrimethylammonium chloride (CTAC), which is available commercially for example as ARQUAD 16-50 ex Akzo.

15

The solubilising agent may optionally further comprise a non-surfactant co-solubiliser. Preferred co-solubilisers include polyethylene glycols (PEG) having a molecular weight ranging from 200 - 6000, most preferably from 1000 - 2000, urea, acid amides up to and including chain lengths of C₆, citric acid and other polycarboxylic acids as disclosed in EP 0 404 471 (Unilever), glycerol, sorbitol, and sucrose. Particularly preferred is propylene glycol.

25

It is advantageous if the weight ratio of solubilising agent (excluding any co-solubiliser present) to hair conditioning compound is in the range of from 1:50 to 1:1, preferably from 1:20 to 1:2, most preferably from 1:10 to 30 1:5.

It is beneficial if the solubilising agent is present at a level greater than about 0.1 wt% of the total composition, such as between 0.1 and 2 wt%, eg between 0.2 and 0.5 wt%.

35

The currently preferred combination of conditioning compound and solubilising agent is 1,2-ditallowoxyloxy-3-trimethylammonium propane chloride (HEQ), conveniently in an amount in the range 1 to 2 wt%, as conditioning compound, and CTAC, conveniently in an amount of about 0.2 wt%, as solubilising agent. Good results have also been obtained by replacing HEQ with dihardened tallow ethyl ester dimethyl ammonium chloride (DEEDMAC), which is available commercially for example as GENAMIN EQ ex Hoechst.

OTHER INGREDIENTS

The hair conditioning compositions of the present invention may contain additional components usually found in hair care compositions.

For instance, the hair conditioning compositions of the invention may contain a suitable amount of a thickening agent such as a polymeric thickener, such as hydroxyethyl cellulose (available commercially as NATROSOL).

One optional component which may be included in the hair conditioning compositions of the invention is a fatty alcohol or fatty acid, or derivative thereof, or a mixture of any of these, having a chain length of from about 8 to about 28 carbon atoms, more preferably from about 12 to about 18 carbon atoms. These materials may be predominantly linear or may be branched.

Such fatty materials may be present in the compositions of the invention in a total amount of from about 0.001 to about 10wt%, more preferably 0.01 to 5wt%, e.g. 0.1 to 1wt% based on the total weight of the composition.

The compositions of the invention may also optionally contain one or more additional conditioning agents.

Suitable additional conditioning agents include cationic polymers, volatile or non-volatile silicones, quaternised 5 silicones (e.g. those materials available under the trade name ABILQUAT ex Goldschmidt), perfluoropolyethers (eg those materials available under the trade name FOMBLIN ex Montefluos), protein hydrolysates, quaternised protein hydrolysates and other materials which are known in the 10 art as having desirable hair conditioning properties..

Additional conditioning agents which are especially suitable include volatile or non-volatile silicone oils, such as for example polyalkylsiloxanes, polyalkylaryl 15 siloxanes, silicone gums, cyclomethicones and aminofunctional silicones. Preferably these silicone materials are incorporated in the compositions as small droplets, preferably of droplet size smaller than 0.1 microns, more preferably smaller than 0.1 microns, most 20 preferably smaller than 0.035 microns. The preferred level of additional conditioning agent(s), if present, in compositions of the invention is up to about 20wt%, for example from 0.01 to 10wt%, more preferably from 0.1 to 5wt% based on the total weight of the composition.

25 Other optional components which may be present in the hair conditioning compositions of the invention in addition to water include perfumes, colouring agents, anti-bacterial agents, anti-dandruff agents, preservatives, proteins, 30 polymers, sunscreens, buffering agents, polyols and other moisturising agents, and natural ingredients such as herb and other plant extracts.

35 The hair conditioning compositions of the invention preferably comprise from 20 to 99.5wt% of water based on the total weight of the composition, preferably 60 to

98wt%, most preferably 75 to 98wt%, so that the composition is in the form of a transparent aqueous dispersion.

5 The composition is conveniently made by agitating a mixture of the hair conditioning compound, solubilising agent and water at a temperature at or above the gel-lamellar phase transition temperature for a suitable time until the formulation becomes transparent, and then adding
10 any further ingredients. For compositions comprising HEQ and CTAC the gel-lamellar phase transition temperature is in the range 45°C to 50°C, and agitation for about 30 minutes at this temperature is appropriate.

15 The hair conditioning compositions of the invention are intended particularly for post-wash use. The composition is applied to wet hair after washing in a suitable amount, eg in the range 5 to 10 grams/head, left for about a minute and then rinsed off.
20 The invention is further illustrated by way of the following non-limitative examples. In the examples all percentages are expressed by weight. Examples of the invention are designated by numbers, whereas Comparative Examples are designated by letters.
25

EXAMPLES

Examples 1 and 2

30 Hair conditioning preparations were prepared containing the following ingredients in the amounts shown below in Table 1.

Table 1

		Example		
		1	2	A
5	INGREDIENT			
	HEQ ⁽¹⁾	1.0	2.0	1.0
	CTAC ⁽²⁾	0.2	0.2	-
	Natrosol ⁽³⁾	1.2	1.2	1.2
	Propylene Glycol (99%)	5.0	5.0	5.0
	Formaldehyde (30%)	0.1	0.1	0.1
	Perfume	0.15	0.15	0.15
		Demineralised water		
		----- to 100 -----		

15 ⁽¹⁾ 1,2 - ditallowoxyloxy - 3- trimethylammonium propane chloride (90% purity).

20 ⁽²⁾ Cetyl trimethyl ammonium chloride, as ARQUAD 16-50 ex Akzo (cationic surfactant).

25 ⁽³⁾ Hydroxyethyl cellulose

Examples 1 and 2 were prepared by adding water to the CTAC and HEQ, heating to 45°C, and stirring at that temperature for about 30 minutes. Once clear, the other formulation ingredients were incorporated at 40-45°C.

30 Comparative Example A was prepared by dissolving HEQ alone in propylene glycol, dispersing the solvent/solid mixture in water at 85°C by fast vortex, and adding the other formulation ingredients during cooling.

Visual Attributes

Clarity of the formulations after mixing was assessed by eye, and the results are given below in Table 2.

Table 2

5

EXAMPLE	APPEARANCE
1	Good clarity with Natrosol and perfume Stable to particle growth/precipitation
2	Good clarity with Natrosol and perfume Stable to particle growth/precipitation
A	Cloudy. Will not solubilise perfume

10

The results show that dispersions of HEQ without CTAC (Comparative Example A) are cloudy. Addition of CTAC, however, (Examples 1 and 2) remedies the problem of cloudiness and allows perfume to be added without inducing cloudiness in the final formulation. Examples 1 and 2 thus result transparent dispersions, having a particle size of less than 0.1 micron. The dispersions of Examples 1 and 2 are stable, and remain transparent, over an extended period of time, of many months.

15

20

Wet Combing Performance

25

The hair conditioning compositions of Examples 1 and 2 were tested against a currently marketed transparent conditioner (Comparative Example B) having the formulation shown below in Table 3:

Table 3

Ingredient	%
Natrosol	1.1
Merquat 100 (60%) ⁽⁴⁾	1.25
Amonyl 380BAU ⁽⁵⁾	2.0
Cremaphore RH410 ⁽⁶⁾	0.6
Sodium chloride	1.0
Perfume	0.3
Minor ingredients & water	to 100

⁽⁴⁾ Polyquaternium-6 (ex Croxton and Garry)

⁽⁵⁾ Cocamidopropyl betaine (30%, ex Seppic)

⁽⁶⁾ PEG 40 Hydrogenated Castor Oil (ex BASF)

15

Table 4 displays a summary of automated wet combing force measurements which provide % reductions in combing force due to conditioner application. Rinse-off conditioner protocols were used in each case.

20

Table 4

Example	% Reduction in Combing Force
1	58
2	60
B	30

30

It can be seen that the Examples of the invention offer a significant improvement in wet combing force reduction over Comparative Example B.

Salon Test

The formulation of Example 2 was salon tested in a paired comparison test against a currently marketed opaque conditioner (Comparative Example C) having the formulation shown below in Table 5:

5

Table 5

	Ingredient	%
	CTAC	2.1
10	Cetearyl alcohol	3.0
	Paraffin wax	1.0
	Glyceryl stearate	0.7
	Formaldehyde (30%)	0.15
	Perfume	0.4
15	Minor ingredients and water	to 100

Various conditioner attributes were hairdresser assessed, and 36 panellists were employed for each comparison. The 20 test protocol used was a 3g dosage each of the respective formulations each per % head in post-wash treatment of wet hair.

The results are shown below in Table 6.

Table 6

Conditioner Attribute	Assessment of Preference
Smooth feel	Equal preference for each formulation
Slippery feel	Comparative Example C preferred
Ease of wet combing	Example 2 preferred
Ease of detangling	Equal preference for each formulation
Gloss	Equal preference for each formulation

10

It can be seen that the formulation of the Example of the invention achieves parity with the formulation of the Comparative Example on all main conditioning attributes despite the Comparative Example's higher proportion of fatty material.

15

Examples 3 and 4Effect of Silicone

20

Hair conditioning preparations were prepared by the method given for Examples 1 and 2 and containing the following ingredients in the amounts shown below in Table 7.

Table 7

	Example	
	3	4
5	HEQ	1.0
	CTAC	0.5
	DC2 - 1685 ⁽⁸⁾	-
	Natrosol	1.2
10	Propylene Glycol (99%)	5.0
	Formaldehyde (30%)	0.1
	Perfume	0.15
	Demineralised water	----- to 100 -----

15 ⁽⁸⁾ silicone microemulsion ex Dow Corning (Dimethiconol (25%)
+ Laureth-23 (6%) + TEA dodecylbenzene sulfonate (15%))

Table 8 displays wet combing results for Examples 3 and 4.

20 Table 8

Example	% Reduction in Combing Force
3	62
4	72

25

It can be seen that inclusion of silicone boosts the wet conditioning attributes of the formulation.

CLAIMS:

1. A hair conditioning composition comprising a transparent dispersion of:

5

(i) a hair conditioning compound comprising a substantially water-insoluble quaternary ammonium material having two C₁₂₋₂₈ alkyl or alkenyl groups connected to the quaternary ammonium head group via at least one ester linkage, and;

10

(ii) a solubilising agent comprising at least one surfactant selected from anionic, amphoteric, nonionic, and cationic surfactants or mixtures thereof.

15

2. A hair conditioning composition according to claim 1, in which the solubilising agent comprises a cationic surfactant.

20

3. A hair conditioning composition according to claim 1 or claim 2, in which the weight ratio of solubilising agent to hair conditioning compound is in the range of from 1:50 to 1:1.

25

4. A hair conditioning composition according to claim 3, in which the ratio of solubilising agent to hair conditioning compound is within the range of from 1:10 to 1:5.

30

5. A hair conditioning composition according to any one of the preceding claims, in which the hair conditioning compound is 1,2 - ditallowoxyloxy - 3 - trimethylammonium propane chloride.

35

6. A hair conditioning composition according to any one of the preceding claims, in which the solubilising agent is cetyl trimethylammonium chloride.
- 5 7. A hair conditioning composition according to any one of the preceding claims, in which the hair conditioning compound is present in an amount in the range 0.01 to 20 wt%.
- 10 8. A hair conditioning composition according to any one of the preceding claims, in which the solubilising agent is present in an amount in the range 0.1 to 2wt%.
- 15 9. A hair conditioning composition according to any one of the preceding claims, which further comprises a volatile or non-volatile silicone.
- 20 10. A hair conditioning composition according to any one of the preceding claims, in which the composition is in the form of self-size-limiting molecular aggregates.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 96/01167

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A61K7/50

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 571 086 (UNILEVER) 24 November 1993 cited in the application see page 5, line 17 - line 30; claims 1,8,9 ---	1,5,6
Y	DATABASE WPI Week 8217 Derwent Publications Ltd., London, GB; AN 82-33766e XP002008574 "Hair rinse conditioner - contg. quaternary ammonium salt, polyoxypropylene and polyoxyethylene for softening effect" & JP,A,57 046 911 (KAO SOAP) , 17 March 1982 see abstract ---	1,5,6 -/-

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents :

- *'A' document defining the general state of the art which is not considered to be of particular relevance
- *'E' earlier document but published on or after the international filing date
- *'L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *'O' document referring to an oral disclosure, use, exhibition or other means
- *'P' document published prior to the international filing date but later than the priority date claimed

*'T' later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

*'X' document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

*'Y' document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

*'A' document member of the same patent family

1

Date of the actual completion of the international search	Date of mailing of the international search report
16 July 1996	26.07.96
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+ 31-70) 340-3016	Authorized officer Voyiazoglou, D

INTERNATIONAL SEARCH REPORT

Int'l Application No PCT/EP 96/01167

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>DATABASE WPI Week 7747 Derwent Publications Ltd., London, GB; AN 77-83933y XP002008575 "Transparent hairdressing soln. comprises a cationic surfactant, a mixt. contg. a neutralised ethoxylated oleyl phosphate ester and water" & JP,A,52 122 638 (UNILEVER) , 15 October 1977 cited in the application see abstract</p> <p>---</p> <p>EP,A,0 636 356 (STEPAN COMPANY) 1 February 1995 see claim 1</p> <p>---</p> <p>EP,A,0 252 441 (HENKEL) 13 January 1988 see claim 1</p> <p>---</p> <p>DE,A,41 38 630 (HENKEL) 27 May 1993 see claim 1</p> <p>---</p> <p>DE,A,24 30 140 (REWO) 19 February 1976 see page 7; claim 1</p> <p>---</p> <p>DATABASE WPI Week 8233 Derwent Publications Ltd., London, GB; AN 82-68940e XP002008576 "Hair cosmetic e. g. rinse, conditioner, lotion etc. - contains quat. ammonium salt, higher alcohol and e. g. hydrogenated rapeseed oil" & JP,A,57 109 707 (LION) , 8 July 1982 see abstract</p> <p>-----</p>	1,6
A		1
A		1
A		1
A		1
A		1
A		1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No	
PCT/EP 96/01167	

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP-A-571086	24-11-93	AU-B-	3706193	28-10-93
		BR-A-	9301619	26-10-93
		CA-A-	2094362	23-10-93
		JP-A-	6009346	18-01-94
		ZA-A-	9302801	21-10-94
EP-A-636356	01-02-95	NONE		
EP-A-252441	13-01-88	DE-A-	3623215	21-01-88
		DE-A-	3774195	05-12-91
		JP-A-	63023846	01-02-88
		US-A-	4874554	17-10-89
DE-A-4138630	27-05-93	DE-D-	59205522	04-04-96
		WO-A-	9310748	10-06-93
		EP-A-	0614349	14-09-94
		ES-T-	2083775	16-04-96
		JP-T-	7509443	19-10-95
DE-A-2430140	19-02-76	NONE		

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- BLACK BORDERS**
- IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- FADED TEXT OR DRAWING**
- BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- SKEWED/SLANTED IMAGES**
- COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- GRAY SCALE DOCUMENTS**
- LINES OR MARKS ON ORIGINAL DOCUMENT**
- REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.